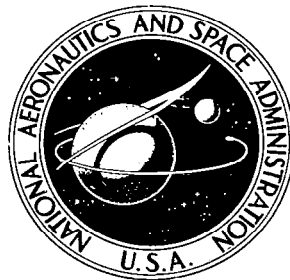


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**OXIDATION OF TD NICKEL AT 1050°  
AND 1200° C AS COMPARED WITH  
THREE GRADES OF NICKEL  
OF DIFFERENT PURITY**

*by Carl E. Lowell, Salvatore J. Grisaffe,  
and Daniel L. Deadmore*

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16. Abstract  The isothermal oxidation of three nickels of different purity, Ni-200, Ni-270, and JM-Ni, was compared with that of TD-Ni in air at 1050° and 1200° C. The samples were oxidized as ground, as polished, or as annealed and polished. Weight change, metal loss, scale thickness, oxide morphology, and scale texture were determined. In degree of oxidation, TD-Ni was nearly the same as the higher purity materials, Ni-270 and JM-Ni; and less pure Ni-200 oxidized more than the others. However, in microstructure and scale texture the TD-Ni more closely resembled Ni-200. Grinding only changed the texture of the oxides of Ni-200 and TD-Ni.					
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# OXIDATION OF TD NICKEL AT 1050<sup>0</sup> AND 1200<sup>0</sup> C AS COMPARED WITH THREE GRADES OF NICKEL OF DIFFERENT PURITY

by Carl E. Lowell, Salvatore J. Grisaffe, and Daniel L. Deadmore

Lewis Research Center

## SUMMARY

The oxidation characteristics of three nickels of different purity, nickel 200 (Ni-200, ~4000 ppm impurity), nickel 270 (Ni-270, ~200 ppm), and Johnson-Mathey spectrographic grade nickel (JM-Ni) were compared with that of thorium oxide dispersion-strengthened nickel (TD-Ni, ~400 ppm with 24 000 ppm ThO<sub>2</sub>). The samples were isothermally oxidized in slowly flowing air at 1050<sup>0</sup> and 1200<sup>0</sup> C. All nickels were oxidized with major surfaces either polished, ground, annealed at 1120<sup>0</sup> C and polished, or annealed at 1240<sup>0</sup> C and polished. Weight change, metal thickness loss, scale thickness, microstructure, and scale texture were examined. In weight change, metal loss, and scale thickness, the TD-Ni was nearly the same as the higher purity grades, JM-Ni and Ni-270, but Ni-200 oxidized approximately twice as much as the others. Grinding and annealing had only second-order effects on these aspects of oxidation behavior.

On the other hand, in scale microstructure and texture TD-Ni was similar to the lower purity Ni-200; Ni-270 was similar to JM-Ni. Grinding did not affect the textures of JM-Ni or Ni-270, which were (001), but it did change the textures of Ni-200 and TD-Ni from (011) to (031). Annealing did not affect the microstructure of the oxide layers. A two-layer scale was observed only on the TD-Ni and Ni-200.

The presence of ThO<sub>2</sub> in TD-Ni appears to act as an impurity in oxide scale orientation but does not substantially alter the extent of oxidation.

## INTRODUCTION

Thorium oxide (ThO<sub>2</sub>) dispersion-strengthened nickel (TD-Ni) has promise for use as a high-temperature turbine vane and combustor material in advanced gas turbine engines. This material is generally coated to extend its life in oxidation, but the influence of ThO<sub>2</sub> on oxidation resistance is important from both fundamental and practical (in case of coating damage) viewpoints.

Several investigators have found that in laboratory furnace testing TD-Ni oxidizes at a slower rate than does pure nickel (refs. 1 and 2). Jones and Westerman (ref. 1) attributed the improved oxidation resistance of TD-Ni to a lowering of the oxide-metal interface reaction rate by the presence of the thorium oxide dispersed phase. However, Wlodek (ref. 2) attributed the improved oxidation resistance of TD-Ni to the high purity of the nickel matrix. Petit and Felton (ref. 3) on the other hand, state that there is essentially no difference between pure nickel and TD-Ni.

Because of these diverse results, a study was conducted on the influence of purity, surface preparation, and high-temperature annealing on the oxidation of various nickel and TD-Ni sheet materials under similar laboratory test conditions. The object of this study was to determine whether the presence of 2.4 percent  $\text{ThO}_2$  in TD-Ni caused it to be more oxidation resistant than pure nickels in a temperature range where its mechanical properties are compatible with aerospace system requirements. To accomplish this purpose, several nickel sheet materials were selected with a wide range of impurity content. They are, in decreasing order of matrix purity: Johnson-Mathey spectrographic grade nickel (JM-Ni, ~30 ppm impurities), Inco nickel 270 (Ni-270, ~200 ppm), TD-Ni (~400 ppm), and Inco nickel 200 (Ni-200, ~4000 ppm). These samples were oxidized isothermally at  $1050^\circ$  and  $1200^\circ$  C for 100 hours. In order to determine the influence of prior material processing, each nickel material was tested with the major surfaces either ground or metallographically polished. Also, specimens of each nickel were annealed at  $1120^\circ$  and at  $1240^\circ$  C to stabilize the grain size and were metallographically polished before oxidation at  $1050^\circ$  C. Degree of oxidation was judged by weight change, scale thickness, and metal recession. Scale morphologies were also compared.

## MATERIALS AND SAMPLE PREPARATION

The chemical analyses for the four nickel materials are shown in table I. Disregarding the  $\text{ThO}_2$  in TD-Ni, Ni-200 is by far the most impure, with Ni-270 and TD-Ni much more pure and nearly the same in total impurity content. Johnson-Mathey nickel is the purest of the starting materials.

All samples were received in sheet form. The thicknesses were nominally: Ni-200, 0.64 centimeter (0.25 in.); Ni-270, 0.13 centimeter (0.05 in.); TD-Ni, 0.13 centimeter (0.05 in.); and JM-Ni, 0.25 centimeter (0.10 in.). The sheets were cut into 2.5-centimeter (1-in.) by 1.9-centimeter ( $3/4$ -in.) specimens. Two surface preparations were used: (1) surface ground with a 600 grit SiC wheel and (2) metallographically polished through 0.5-micrometer ( $\mu\text{m}$ ) diamond paste. Part of the samples were annealed before polishing. The annealing was carried out under a hydrogen atmosphere at  $1120^\circ$  C for 1 hour with some samples and  $1240^\circ$  C for 2 hours with others. All samples were cleaned in methanol just before oxidation.

TABLE I. - CHEMICAL ANALYSES OF NICKEL MATERIALS

[All values in ppm.]

Element	Nickel 200	Nickel 270	Johnson-Mathey nickel	TD-Ni
Ni	Bal.	Bal.	Bal.	Bal.
C	600	113	17	40
Mn	2700	<10	---	---
Fe	400	10	3	<100
Cu	100	20	1	50
Cr	----	<10	---	100
S	50	<5	---	34
Si	200	<10	3	---
Mg	----	<10	2	---
Ti	----	---	---	<10
Co	----	10	---	<100
Al	----	<10	1	---
Ca	----	---	1	---
Ag	----	---	1	---
ThO <sub>2</sub>	----	---	---	24 000
Total impurity	4050	<208	29	< 434 >24 000 with ThO <sub>2</sub>

## EQUIPMENT AND PROCEDURES

## Gravimetric Analysis

The weight-gain oxidation apparatus is shown schematically in figure 1. The specimens, after preparation, were suspended above the furnace from one pan of the balance. Weights were added to the other pan to attain a null reading. After the temperature of the furnace was stabilized at the oxidation temperature, the furnace was raised to position the sample in the center of the hot zone. The recorder was then started and remained on for the duration of the 100-hour runs to record weight change ( $\Delta W$ ). The accuracy of the recorded weights was  $\pm 0.5$  milligram. After a slight initial drop in temperature when the furnace was raised, the holding temperature was regained in less than 10 minutes. The temperature remained constant from then on to within  $\pm 2^\circ \text{C}$ . The airflow through the furnace was by convection only.

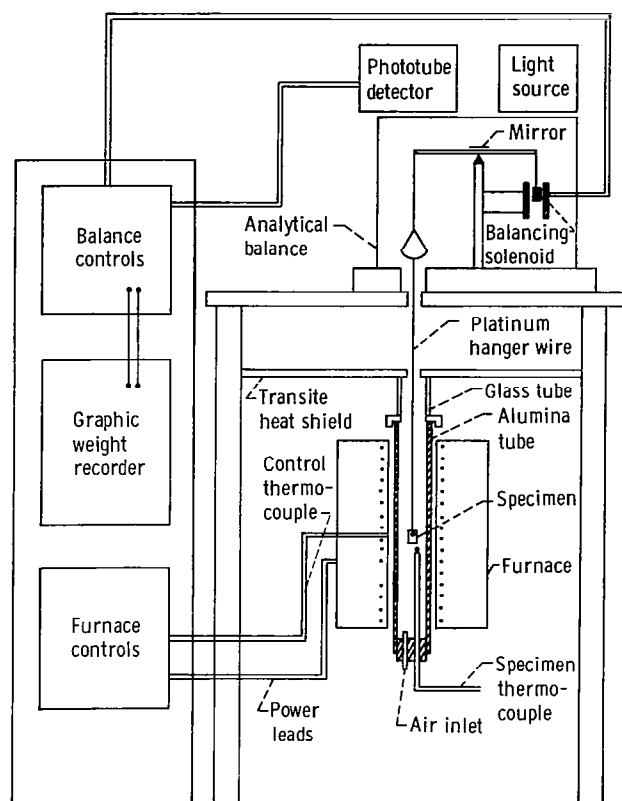


Figure 1. - Continuous weight-gain apparatus.

## Metal Loss and Scale Thickness

Before oxidation, the thicknesses of all samples were measured with a micrometer to a precision of  $\pm 2$  micrometer. A series of measurements were made on each sample along the centerline to assess the parallelism of the major faces. After oxidation, the samples were mounted in epoxy and sectioned as nearly as possible along the centerline previously measured. The remaining metal thickness and scale thickness measurements were made at approximately the same locations used originally. These measurements were made with a filar eyepiece with a precision of  $\pm 2$  micrometers.

## Metallography

The oxidized surfaces of all samples were examined at  $\times 250$ . The specimens were then mounted, sectioned, and polished. The nickel substrates were etched with 93 HCl:  $2\text{HNO}_3:5\text{H}_2\text{SO}_4$ . After viewing and photographing the samples, they were repolished,

and then the NiO scales were electrolytically etched using a solution of 1  $\text{CH}_3\text{COOH}$ : 1HF:4 $\text{H}_2\text{O}$  (a technique developed by Dr. J. Wolf (ref. 4)).

## Pole Figures

A pole figure diffractometer was used to determine the (111) pole figure of both the as-prepared and as-oxidized specimens. Nickel filtered copper radiation was used for the Schultz method (ref. 5). The intensity distribution was scanned with a proportional counter along a helical path over a  $360^\circ$  azimuthal range and  $0^\circ$  to  $80^\circ$  inclination. Since no randomly oriented, high density NiO was available, no corrections, other than for background, were made.

## RESULTS AND DISCUSSION

### Gravimetric Analysis

The results of the weight-change experiments are plotted in figures 2 to 4 and are summarized in table II. The weight change ( $\Delta W$ ) against time curves (see figs 2 to 4)

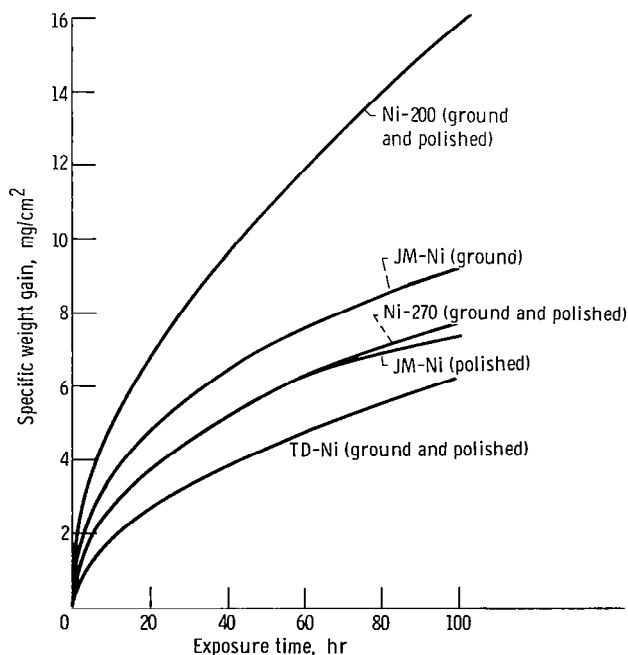


Figure 2. - Oxidation of various nickels at  $1050^\circ\text{C}$ .

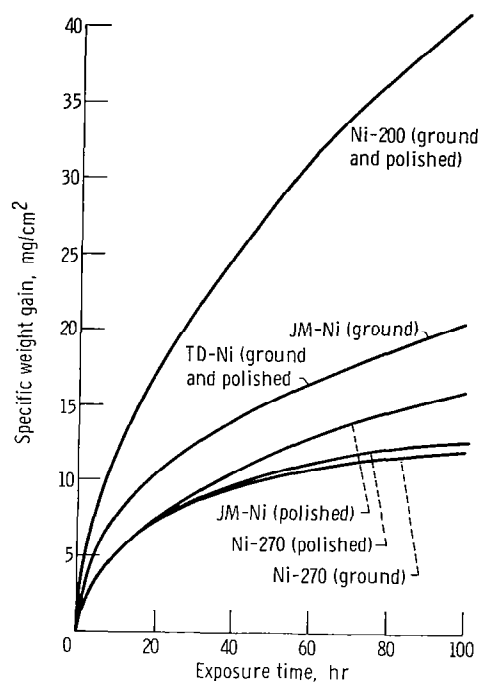


Figure 3. - Oxidation of various nickels at 1200° C.

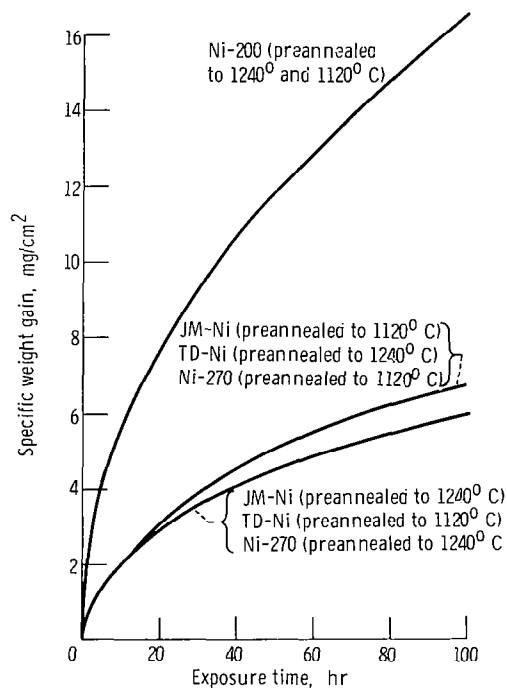


Figure 4. - Oxidation of various nickels at 1050° C after annealing to 1120° and 1240° C.



TABLE II. - WEIGHT GAIN IN AIR IN 100 HOURS

Conditions	Ni-200	Ni-270	JM-Ni	TD-Ni
	Weight gain, $\Delta W$ , mg/cm <sup>2</sup>			
1200° C - ground	<sup>a</sup> 41.0	<sup>a</sup> 13.1	19.9	15.8
1200° C - polished	<sup>a</sup> 40.4	<sup>a</sup> 13.8	14.3	15.8
1050° C - ground	<sup>a</sup> 15.8	<sup>a</sup> 7.8	9.2	6.2
1050° C - polished, no anneal	<sup>a</sup> 15.6	<sup>a</sup> 8.1	7.6	6.2
Annealed to 1120° C - 1 hr in hydrogen	<sup>a</sup> 16.5	<sup>a</sup> 6.9	<sup>a</sup> 6.9	6.2
Annealed to 1240° C - 2 hr in hydrogen	<sup>a</sup> 16.5	<sup>a</sup> 5.8	<sup>a</sup> 6.0	<sup>a</sup> 6.8

<sup>a</sup>Grain boundary attack observed metallographically.

show clearly that under all conditions Ni-200 gains the most weight. Although TD-Ni showed the lowest weight gains of all materials tested in either the ground or polished conditions at 1050° C, this was not the case at 1200° C. Here, Ni-270 (~200 ppm) gained less while JM-Ni (~30 ppm) and TD-Ni (~400 ppm) showed intermediate weight gains. Annealing at 1120° C and polishing produced about the same ranking as polishing alone in the 1050° C tests. Annealing at 1240° C and testing at 1050° C (polished specimens) resulted in the same ranking as the 1200° C tests.

Although grinding before oxidation increased the 100-hour weight change ( $\Delta W$ ) of JM-Ni by approximately one-third, as compared with polished samples, it has little or no effect on the  $\Delta W$  of the other nickels. Annealing seemed to decrease the  $\Delta W$  of Ni-270 and JM-Ni, but it increased that of TD-Ni and Ni-200 slightly if at all.

The substantially higher weight gains of the Ni-200 are probably due to the high manganese content. Evans et al. (ref. 6) have shown that small amounts of manganese increase the rate of oxidation of nickel. Also (as indicated by the footnoted values in table II), Ni-200 and Ni-270 showed some grain boundary attack. The additional area for oxygen reaction might account for some of the higher weight gain in Ni-200, but it does not relate to the low gain on Ni-270.

In addition to the linear plots shown in figures 2 to 4, plots of  $(\Delta W/A)^2$  against time were made (not shown) to see if the curves were parabolic. In many cases such plots were not simple straight lines, but appeared to consist of an initial and a final straight-line segment, and a nonlinear transition period between them. Most of such transitions were abrupt, but some lasted many hours. Table III gives both the initial and final apparent parabolic rate constants  $K_p$  from the linear portions of  $(\Delta W/A)^2$  against  $t$

TABLE III. - PARABOLIC RATE CONSTANT DATA ON FOUR NICKELS

Oxida- tion tem- perature. °C	Material	Material condition	Approximate initial para- bolic rate constant, $K_p$ , mg/(cm <sup>4</sup> )(hr)	Approximate time at which initial $K_p$ changed, hr	Approximate final $K_p$ , mg/(cm <sup>4</sup> )(hr)
1200	Ni-200	Ground	16.6	No change	----
		Polished	15.2	No change	----
	Ni-270	Ground	2.6	15	1.6
		Polished	2.6	15	1.7
	J-M Ni	Ground	6.0	10 - 50	3.3
		Polished	2.6	15 - 70	2.1
	TD-Ni	Ground	2.7	No change	----
		Polished	2.7	No change	----
1050	Ni-200	Ground	2.6	No change	----
		Polished	2.5	No change	----
		Anneal at 1120° C	2.8	No change	----
		Anneal at 1240° C	2.5	No change	----
	Ni-270	Ground	0.8	25	0.53
		Polished	.8	25	.62
		Anneal at 1120° C	.55	10	.27
		Anneal at 1240° C	.58	10 - 40	.46
	J-M Ni	Ground	1.2	10 - 60	0.68
		Polished	.72	25	.53
		Anneal at 1120° C	.48	No change	----
		Anneal at 1240° C	.73	10	.32
	TD-Ni	Ground	0.39	No change	----
		Polished	.39	No change	----
		Anneal at 1120° C	.39	No change	----
		Anneal at 1240° C	.62	5	0.47

curves and the approximate time at which the nonlinear transition periods began. Both TD-Ni (~400 ppm) and Ni-200 (~4000 ppm) had but one  $K_p$  for the entire 100 hours under all conditions except for the 1240° C annealed TD-Ni. Just the reverse is true for the other two nickels; both had transition periods (some for as long as 55 hr) except the 1120° C annealed JM-Ni (~30 ppm). Since, at least for the nonannealed specimens, neither TD-Ni or JM-Ni exhibited grain boundary attack, the changes in  $K_p$  observed for JM-Ni (and Ni-270) do not appear related to this phenomenon.

## Oxide Scale Thickness and Metal Loss

These data are presented in table IV and must be interpreted with some care. The metal loss data ( $\Delta t$ ) would be the more significant measurements if internal oxidation had not taken place. However, all of the nickels oxidized showed internal oxidation or grain boundary attack under some conditions (see the section Metallography). In these instances scale thickness is a better criterion as long as volatilization of NiO is not appreciable, which was the case here. (NiO volatilization loss was checked by comparing  $\Delta W$  with  $\Delta t$  where no internal oxidation had occurred. Calculations of expected metal loss due to the formation of NiO were made assuming the  $\Delta W$  was a result of oxygen pickup. These calculations were within about 5 percent agreement with the  $\Delta t$ . Therefore, little, if any, volatilization of NiO occurred.)

Table IV shows that, on the basis of either oxide scale or thickness loss, Ni-200 (~4000 ppm impurity) is the least oxidation resistant and TD-Ni (~400 ppm) is generally not more oxidation resistant than the JM-Ni (~30 ppm) or Ni-270 (~200 ppm).

While grinding before oxidation did not markedly affect the behavior of TD-Ni at either temperature, it appears that the influence of grinding (as compared with polishing) on the other materials before exposure is related more to temperature than to purity.

TABLE IV. - OXIDE SCALE THICKNESSES AND METAL THICKNESS LOSSES  
AFTER VARIOUS OXIDATION EXPOSURES

Conditions	Ni-200		Ni-270		JM-Ni		TD-Ni	
	Oxide scale thick-ness. $\mu m$	Metal loss. $\mu m$	Oxide scale thick-ness. $\mu m$	Metal loss. $\mu m$	Oxide scale thick-ness. $\mu m$	Metal loss. $\mu m$	Oxide scale thick-ness. $\mu m$	Metal loss. $\mu m$
1200° C - ground	305	286	112	93	112	117	136	135
1200° C - polished	301	320	123	86	105	95	133	132
1050° C - ground	110	108	66	50	81	69	60	51
1050° C - polished, no anneal	108	104	68	63	57	58	65	47
Annealed to 1120° C - 1 hr in hydrogen	116	122	71	45	62	49	61	53
Annealed to 1240° C - 2 hr in hydrogen	125	117	63	29	44	17	62	41

Grinding followed by oxidation at 1050° C decreased the metal loss of Ni-270 but increased that of JM-Ni; it had an erratic influence on oxide scale thickness. At 1200° C, prior grinding decreased the metal loss of Ni-200 but increased that of JM-Ni and Ni-270.

Annealing the samples before oxidation produced a change in the relative extent of oxidation shown for the unannealed, polished specimens in table IV. Although the metal loss and scale thickness for Ni-200 increased, those on JM-Ni and Ni-270 decreased after annealing at either temperature but more significantly after the 1240° C anneal. The metal loss of TD-Ni was also decreased after the 1240° C anneal but increased after the 1120° C anneal, while the scale thickness remained approximately constant.

## Metallography

Metallographic cross sections of Ni-200, Ni-270, JM-Ni, and TD-Ni are shown after the various oxidation exposure conditions in figures 5 to 8, respectively. In these figures, the nickel substrate has been etched. In figures 9 and 10 etched oxide scales are shown of samples with polished surfaces tested at 1050° and 1200° C, respectively.

The Ni-200 (~4000 ppm impurities) was susceptible to both grain boundary and internal oxidation in all cases (see fig. 5). This is probably due to the preferential oxidation of the manganese impurity. Etching of the scale showed a two-layer structure (see figs. 9 and 10). The outer layer consisted of large columnar grains with small equiaxed grains in the inner layer. The difference in grain size in the two layers is substantial.

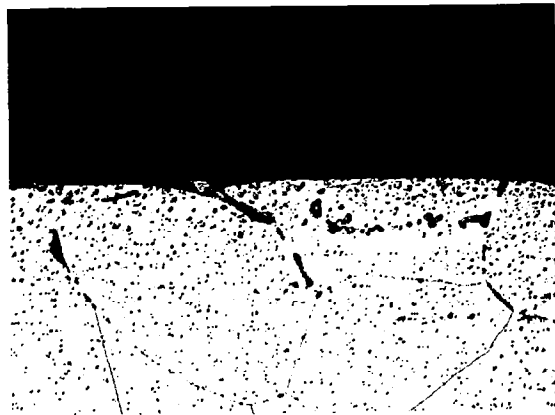
Nickel-270 (~200 ppm impurities) (fig. 6) shows grain-boundary oxidation under all conditions. The oxidation was probably due to accumulation of impurities at the grain boundaries. The etched scale is shown to have only one layer (figs. 9 and 10). This is in contrast to several investigators (that is, ref. 8) who have observed a two layered NiO on this material.

Examination of the microstructures of oxidized JM-Ni (~30 ppm impurities) revealed no internal oxidation in any of the unannealed samples (see fig. 7). Annealing at 1120° C before oxidation resulted in grain-boundary oxidation; annealing at 1240° C before oxidation resulted in both grain-boundary oxidation and internal oxidation. Etching the oxide formed at either temperature (figs. 9 and 10) shows only a single-layered scale. The oxide appears to consist of equiaxed, large NiO grains that were slightly smaller than those observed on Ni-270.

The microstructure of TD-Ni (~400 ppm impurities) shows no evidence of grain-boundary oxidation except after the 1240° C anneal (fig. 8). However, the etched scale revealed two layers as did Ni-200 (see figs. 9 and 10). The outer layer appears to be columnar with the inner layer consisting of smaller equiaxed grains. The grain size difference is smaller between the two layers than observed in Ni-200.



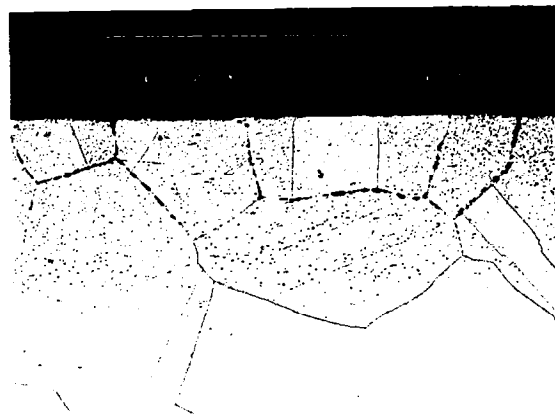
(a) Oxidized at 1200° C, ground surface.



(b) Oxidized at 1200° C, polished surface.



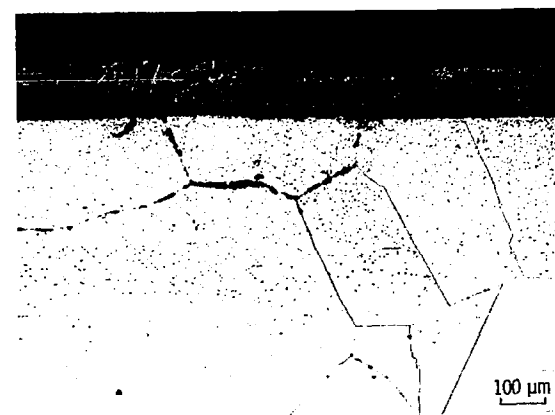
(c) Oxidized at 1050° C, ground surface.



(d) Oxidized at 1050° C, polished surface.

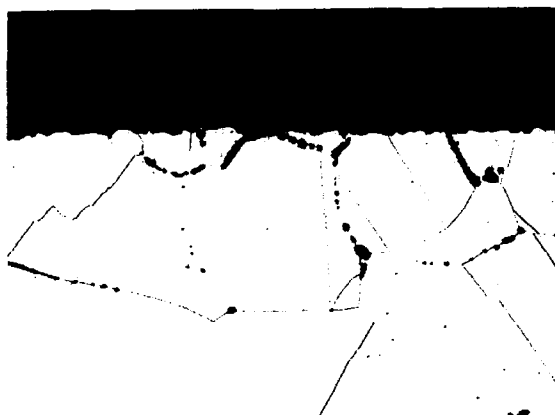


(e) Oxidized at 1050° C; polished surface; annealed to 1120° C.

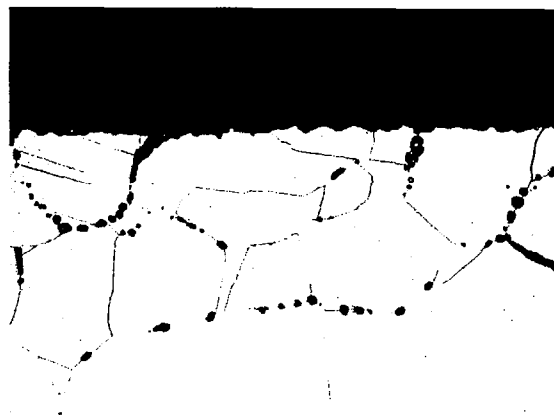


(f) Oxidized at 1050° C; polished surface; annealed to 1240° C.

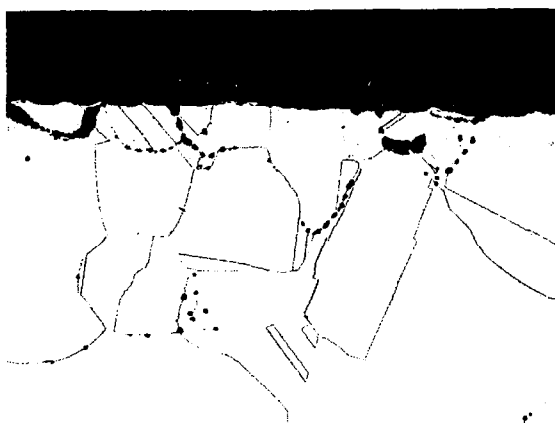
Figure 5. - Microstructures of oxidized Ni-200; X100.



(a) Oxidized at 1200° C, ground surface.



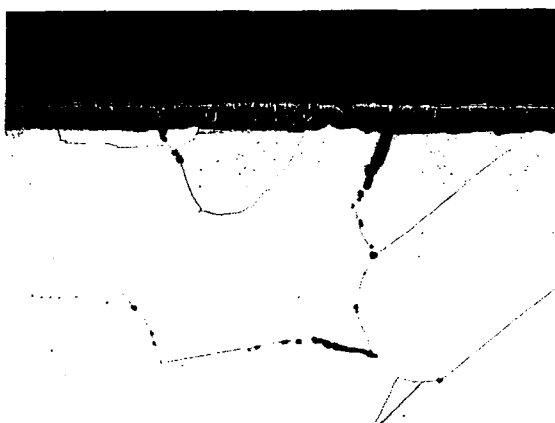
(b) Oxidized at 1200° C, polished surface.



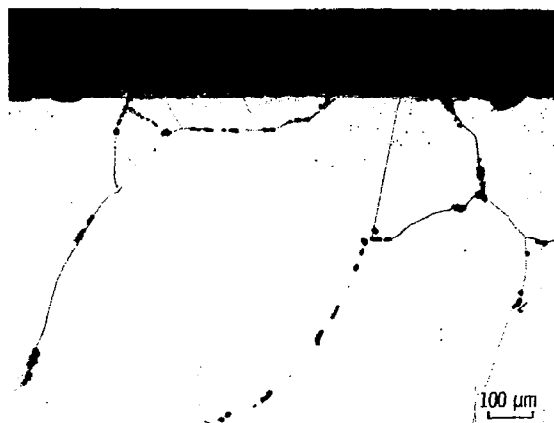
(c) Oxidized at 1050° C, ground surface.



(d) Oxidized at 1050° C, polished surface.

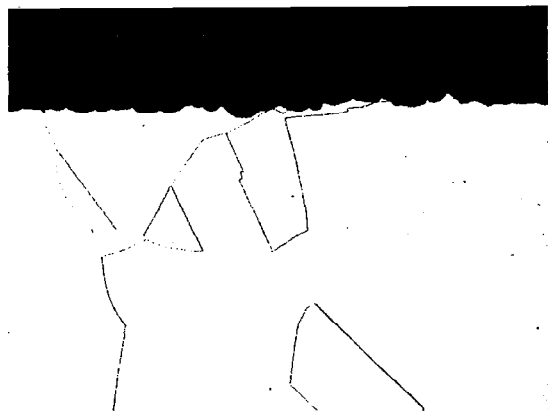


(e) Oxidized at 1050° C; polished surface; annealed to 1120° C.

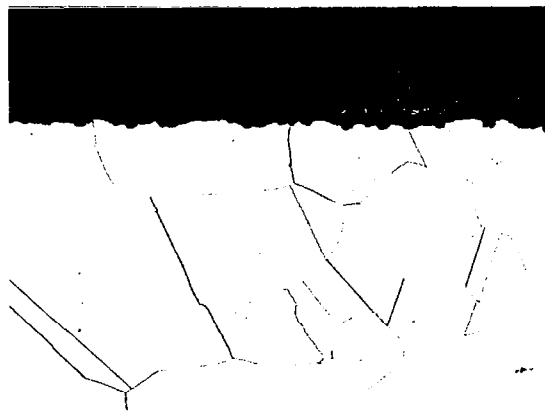


(f) Oxidized at 1050° C; polished surface; annealed to 1240° C.

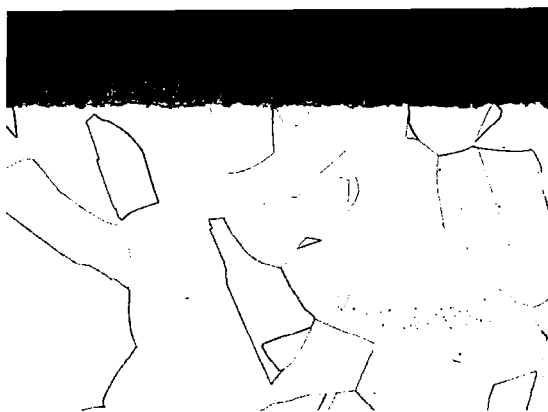
Figure 6. - Microstructures of oxidized Ni-270; X100.



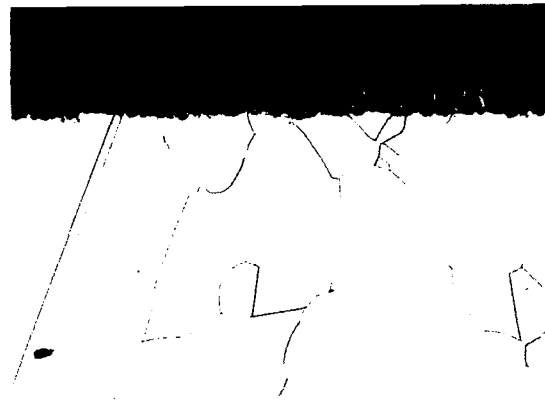
(a) Oxidized at 1200° C, ground surface.



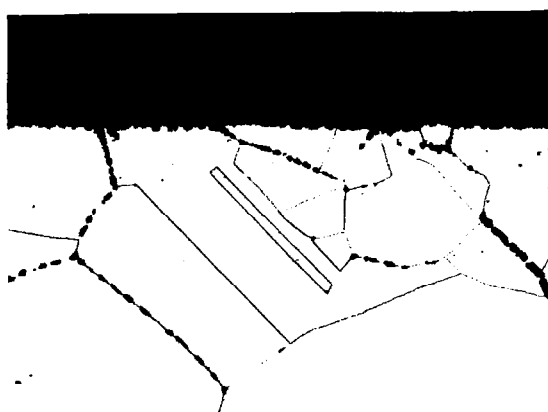
(b) Oxidized at 1200° C, polished surface.



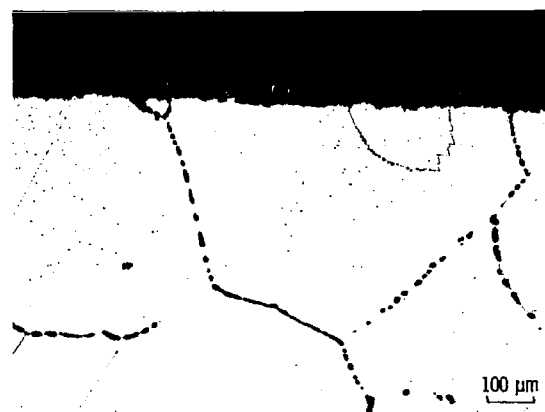
(c) Oxidized at 1050° C, ground surface.



(d) Oxidized at 1050° C, polished surface.

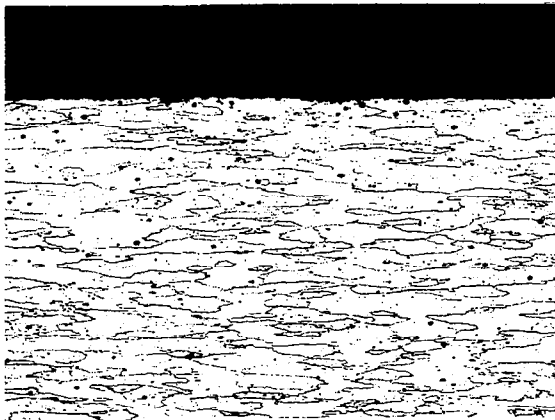


(e) Oxidized at 1050° C; polished surface; annealed to 1120° C.

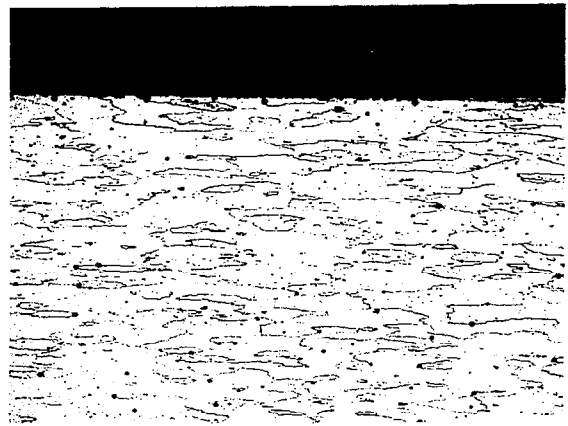


(f) Oxidized at 1050° C; polished surface; annealed to 1240° C.

Figure 7. - Microstructures of oxidized spectrographically pure nickel (JM-Ni); X100.



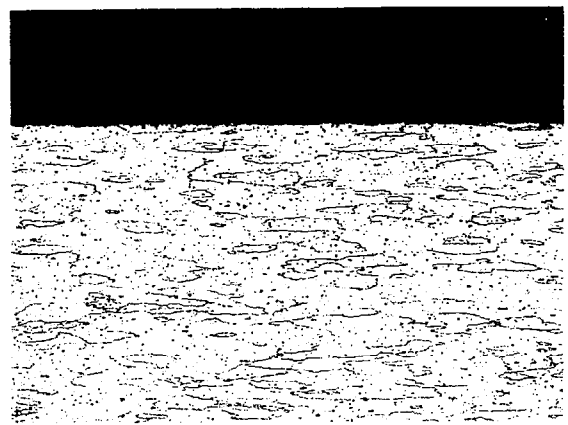
(a) Oxidized at 1200° C, ground surface.



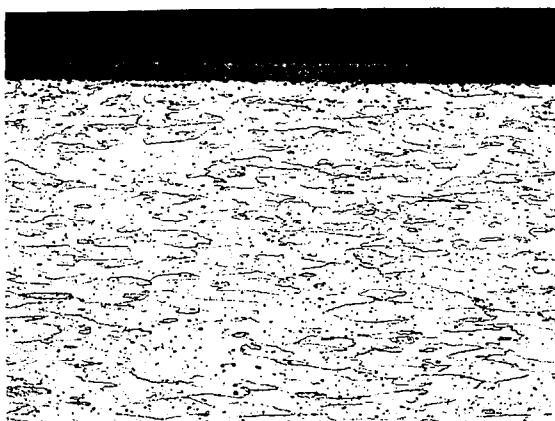
(b) Oxidized at 1200° C, polished surface.



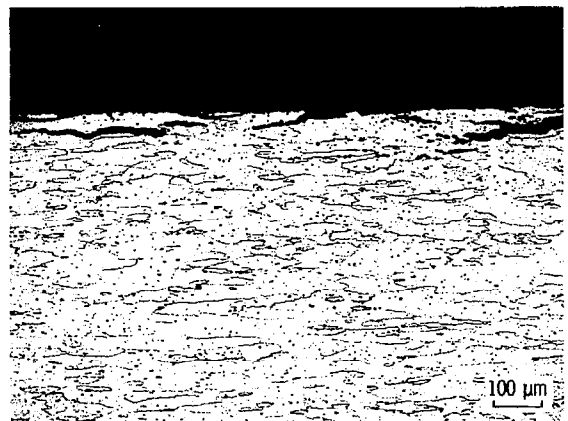
(c) Oxidized at 1050° C, ground surface.



(d) Oxidized at 1050° C, polished surface.



(e) Oxidized at 1050° C; polished surface; annealed to 1120° C.



(f) Oxidized at 1240° C; annealed; polished surface.

Figure 8. - Microstructures of oxidized TD-Ni; X100.

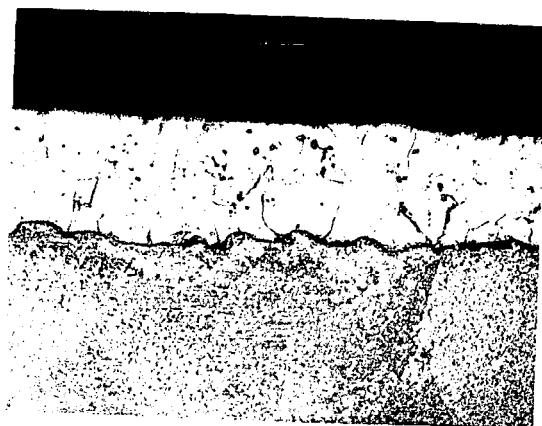




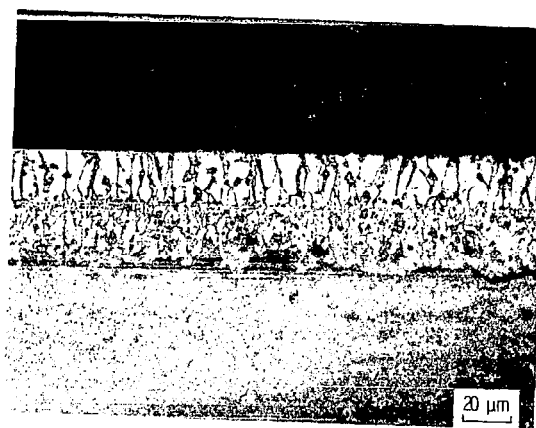
Ni-200



Ni-270

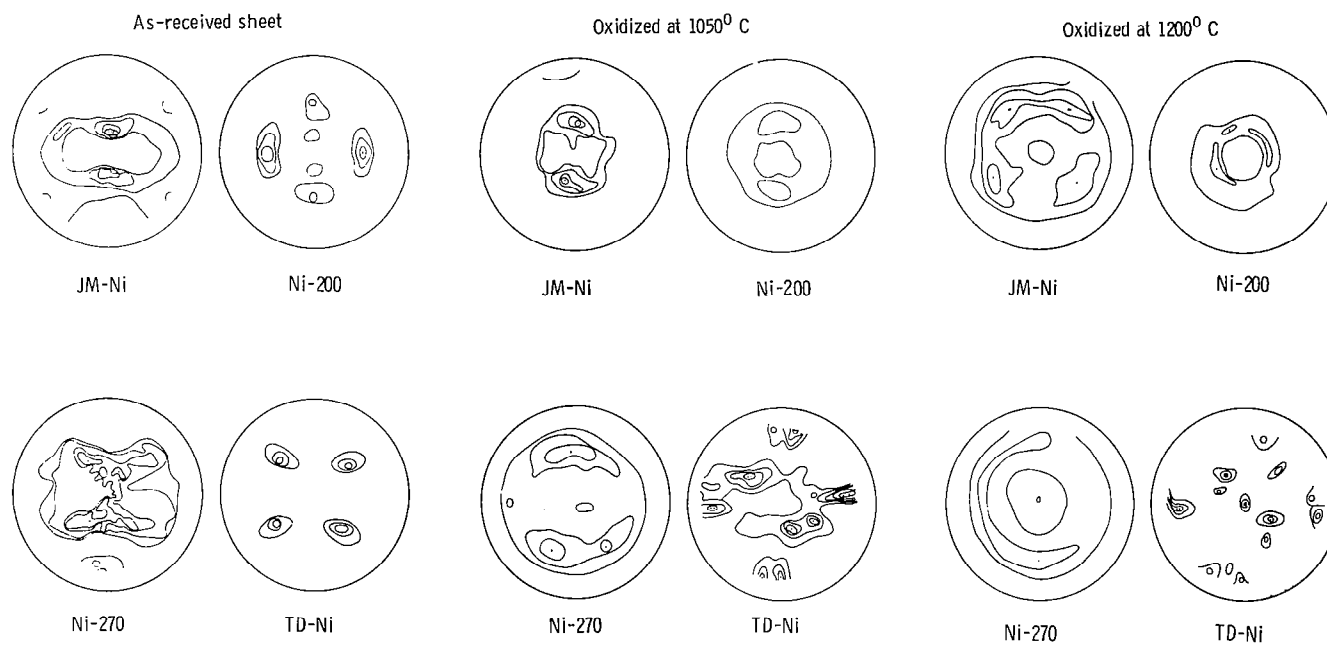


JM-Ni



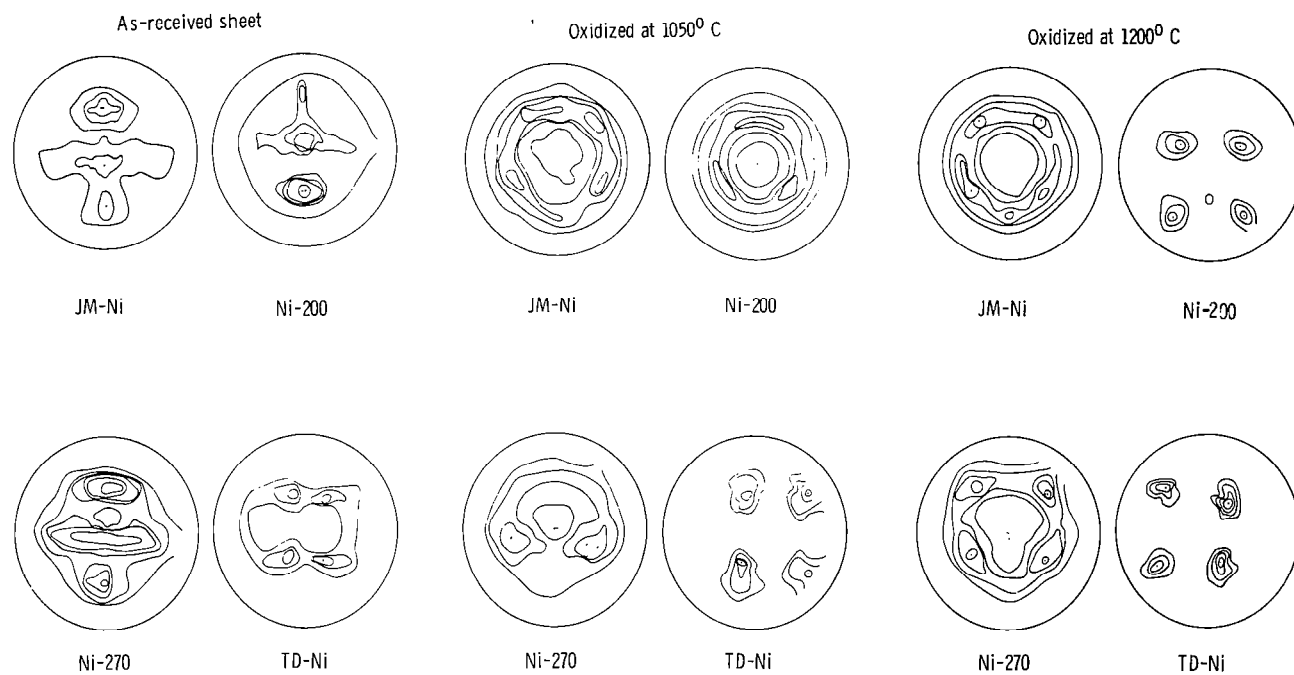
TD-Ni

Figure 9. - Etched oxide scales on nickel substrates polished before oxidation for 100 hours at 1050° C; X500.



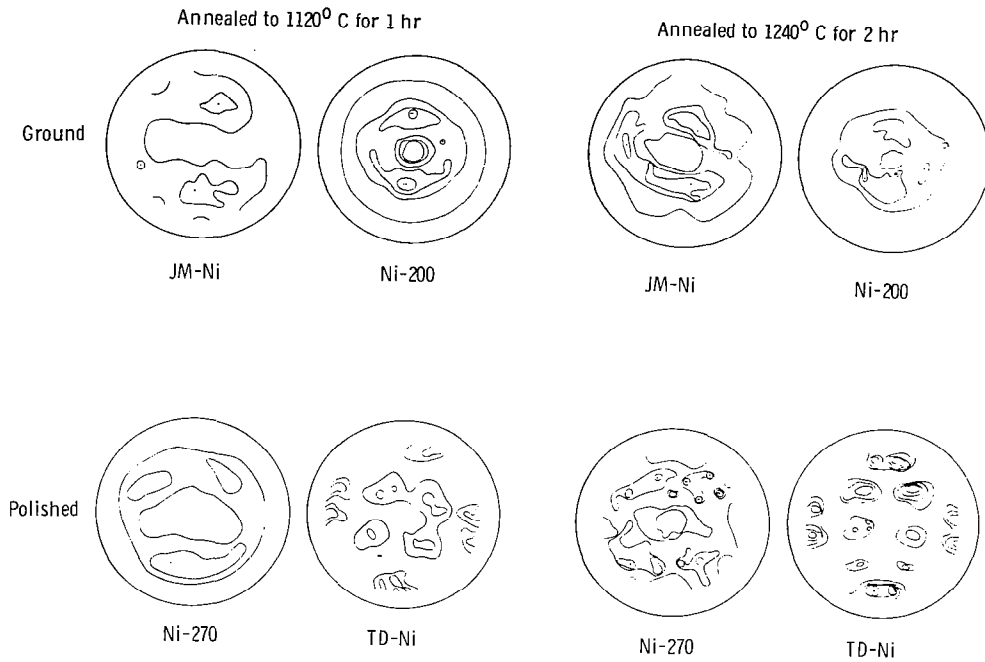
(a) All surfaces polished before oxidation; (111) pole.

Figure 12. - Pole figures of the surface oxides formed on TD-Ni and three nicks of different purities.



(b) All surfaces ground before oxidation; (111) pole.

Figure 12, - Continued.



(c) All surfaces annealed before oxidation at 1050° C; (111) pole.

Figure 12. - Concluded.

TABLE V. - TEXTURE SUMMARY

Type of material	Conditions	Ni-200		Ni-270		JM-Ni		TD-Ni	
		Plane	Direction	Plane	Direction	Plane	Direction	Plane	Direction
Oxide	1200° C - ground	(031)	[100]	(001)	[100]	(001)	[100]	(031)	[100]
	1200° C - polished	(011)	Random	(001)	Random	(001)	[100]	(011)	[21 $\bar{1}$ ] <sup>b</sup>
	1050° C - ground	(031)	[100]	(001)	[100]	(001)	[100]	(031)	[100]
	1050° C - polished, no anneal	(011)	[100]	(001)	Random	(001)	[100]	(011)	[21 $\bar{1}$ ] [1 $\bar{1}$ 1]
	Annealed at 1120° C - 1 hr in hydrogen	(011)	[100]	(001)	Random	(001)	Random	(011)	[21 $\bar{1}$ ] [1 $\bar{1}$ 1]
	Annealed at 1240° C - 1 hr in hydrogen	(011)	[100]	(001)	Random	(001)	Random	(a)	(a)
Metal	As-received ground	Random	[313]	Random	[313]	Random	[313]	(031)	[100] <sup>b</sup>
	As-received polished	(001)	[110]	(112)	[11 $\bar{1}$ ] <sup>b</sup>	(112)	[11 $\bar{1}$ ]	(001)	[100]

<sup>a</sup>Undefined.

<sup>b</sup>Weak secondary texture.

Thus, the orientation of the oxide scale seems to be affected by the previous condition of the metal only for Ni-200 and TD-Ni - the two metals that oxidize to a duplex scale. These orientations do not, however, correspond to the epitaxial relations developed by Cathcart et al. (ref. 7).

## SUMMARY OF RESULTS

A study was conducted to compare the weight gain, metal loss, oxide thickness, and oxide morphology of three nickel materials of different purity with thorium dispersion-strengthened nickel (TD-Ni) after a 100-hour isothermal oxidation at 1040° and 1200° C. Grinding, polishing, and hydrogen annealing at 1120° C (1 hr) and 1240° C (2 hr) were used to examine the influence of prior treatment. The three nickel materials included Ni-200 (~4000 ppm impurities), Ni-270 (~200 ppm), and JM-Ni (~30 ppm).

Ni-200, the most impure nickel, gained the most weight under all conditions. The weight changes of the other three nickels were quite close to one another, but substantially lower than the Ni-200. TD-Ni was slightly better than Ni-270 or JM-Ni except at 1200° C where it was slightly worse. The kinetics of weight change grouped the nickels differently: TD-Ni and Ni-200 oxidized parabolically while Ni-270 and JM-Ni did not.

Metal recession and scale thickness data gave results similar to the weight-gain data. Nickel-200 showed the greatest amount of oxidation; TD-Ni, JM-Ni, and Ni-270 were similar, with TD-Ni showing the least amount of oxidation except at 1200° C where it was slightly worse than Ni-270 and JM-Ni.

The microstructures of the oxides on TD-Ni and Ni-200 consisted of two layers. The layer at the oxide metal interfaces was composed of small, equiaxed grains, the outer layer was large grained and columnar. In contrast, the microstructure of the oxides on JM-Ni and Ni-270 consisted of a single layer of nearly equiaxed grains.

In preferred orientation, the oxides on TD-Ni and Ni-200 were also alike. Both were (110) except on the ground samples which were (031). The orientation of the oxides on JM-Ni and Ni-270 was always (001).

## CONCLUSIONS

The oxidation behavior of thorium oxide dispersion-strengthened nickel (TD-Ni) was compared with nickel 200, nickel 270, and Johnson-Mathey spectrographic grade nickel at 1200° and 1050° C. The oxidation behavior was judged by weight gain scale thickness, metallography, and pole figure analysis. From this work the following conclusions were drawn:

1. Since the weight change, scale thickness, and metal loss data were similar for TD-Ni (~4000 ppm impurities), nickel 270 (200 ppm), and Johnson-Mathey spectrographic grade nickel (~30 ppm), and were all much less than nickel 200 (~4000 ppm), the presence of 24 000 ppm thorium dioxide has little or no impurity effect on the growth rate of nickel oxide.

2. The thorium oxide in TD-Ni has a marked effect on the morphology of the nickel oxide scale. In this respect it acts like the impurities in nickel 200 (~4000 ppm) in that both materials form dual layered scales of similar orientation.

3. Surface preparation by polishing or grinding and preoxidation annealing have only second-order effects on the oxidation rate of the nickels studied, although these treatments can affect the oxide orientation of TD-Ni and nickel 200.

Lewis Research Center,  
National Aeronautics and Space Administration,  
Cleveland, Ohio, October 15, 1971,  
134-03.

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